

CERIUM ORGANIC COLLOIDAL DISPERSION AND ELEMENT SELECTED  
FROM RHODIUM AND PALLADIUM AND USE THEREOF AS AN ADDITIVE  
TO DIESEL FUEL FOR INTERNAL COMBUSTION ENGINES

5       The present invention relates to an organic colloidal dispersion of cerium and of an element selected from rhodium and palladium, and the use thereof as a diesel fuel additive for internal combustion engines.

10      It is known that during the combustion of diesel fuel in the diesel engine, the carbonaceous products tend to form soot, which is considered to be harmful both to the environment and to health. Research has been conducted for many years on techniques for reducing the emission of these carbonaceous particles.

15      One satisfactory solution that is now used in mass-produced engines consists in collecting the particles on a filter that is regularly regenerated to prevent the clogging thereof. Filter regeneration is easier if the autoignition temperature of the soot is low, and this can be obtained by introducing a catalyst into the core of the soot during combustion. This technology, known as "Fuel Borne Catalysis" or FBC, is also widely used. The soot thereby containing additives has an autoignition temperature that is sufficiently low to be reached 20 frequently during the normal running of the engine or 25 during specific regeneration cycles.

It is known that dispersions of rare earth compounds, used as fuel additives, can serve to reduce the autoignition temperature of soot.

30      Furthermore, it is also important to limit the emission of the nitrogen oxides ( $\text{NO}_x$ ) by diesel engines.

Hence a real need remains for an effective catalyst for both lowering the soot combustion temperature and reducing the  $\text{NO}_x$  content of the diesel

engine exhaust gases.

It is the object of the invention to provide such a catalyst.

For this purpose, the colloidal dispersion of  
5 the invention is of the type comprising particles of a cerium compound, an acid and an organic phase, and is characterized in that it further comprises a compound of at least one element selected from rhodium and palladium.

10 According to a first variant of the invention, the colloidal dispersion is characterized in that it comprises particles of a compound of cerium and of another rare earth.

15 According to a further variant, the colloidal dispersion of the invention is characterized in that it comprises particles based on a compound of cerium, optionally of another rare earth, and of iron.

20 Other features, details and advantages of the invention will appear even more completely from a reading of the description that follows, and of concrete but nonlimiting examples provided to illustrate it.

25 For the rest of the description, rare earth means elements of the group consisting of yttrium and the elements of the Periodic Table with atomic numbers from 57 to 71 inclusive.

The expression "colloidal dispersion" in the present invention designates any system consisting of fine solid particles of colloidal size based on a  
30 cerium compound and, according to the abovementioned variants, of a rare earth other than cerium and/or iron, in suspension in a liquid phase, said particles further optionally containing residual quantities of bound or adsorbed ions such as, for example, nitrates, acetates, citrates and ammoniums. Colloidal size means size between about 1 nm and about 500 nm. The particles may more particularly have an average size of  
35 not more than about 250 nm, particularly of not more

than 100 nm, preferably of not more than 20 nm, and even more preferably of not more than 15 nm. In such dispersions, the cerium, the other rare earth and/or the iron may be present either, preferably, completely 5 in colloidal form, or in colloidal form and partially in ionic form.

More particularly, if the particles of the dispersion of the invention are based on a compound of a plurality of elements, that is, cerium, another rare 10 earth and/or iron, these elements are mixed within each particle, and generally have the form of mixed oxides and/or hydrated mixed oxides (oxyhydroxides).

The cerium is mainly in the form of cerium IV. For example, the cerium III content with respect to 15 cerium IV (content expressed by the atomic ratio Ce III/Ce total) is generally not more than 40%. It may vary according to the embodiments of the dispersions used and may thus be not more than 20%, more particularly not more than 10%, and even more 20 particularly not more than 1%.

In the case of the abovementioned first variant, the rare earth other than cerium may be more particularly lanthanum or praseodymium. Obviously, the present variant covers the case in which the particle 25 is a compound of cerium and of a plurality of other rare earths in combination.

The proportion of rare earth other than cerium is preferably at least 10%, more particularly at least 20% and even more particularly not more than 50%, in 30 moles with respect to the total number of moles of cerium and of rare earth expressed as oxide.

In the case of the second variant, the proportion of cerium is preferably not more than 50%, more particularly not more than 20% and even more 35 particularly not more than 10%, this proportion being expressed as moles of cerium oxide  $\text{CeO}_2$  with respect to the total number of moles of cerium oxide and of iron oxide  $\text{Fe}_2\text{O}_3$ .

The two variants may be combined, that is, the particles may be compounds of cerium, of at least one other rare earth, and of iron.

According to the main feature of the invention,  
5 the colloidal dispersion further contains a compound of at least one element selected from rhodium and palladium. The invention applies in particular to the case in which rhodium and palladium are present in combination. Palladium has the additional effect of  
10 favoring the oxidation of CO and unburnt hydrocarbons in exhaust gases.

Generally, the rhodium and/or palladium content is not more than 5%, more particularly not more than 1% and even more particularly not more than 0.5% with  
15 respect to the combination of the elements cerium, other rare earth and iron in the particles. This content is expressed as a % by weight of rhodium and/or palladium metal with respect to the sum of the weights of the elements cerium, rare earth and iron. The upper limit for the rhodium and/or palladium content is not critical, but simply of an economic order, because an excessive quantity of these elements incurs a higher cost of the dispersion without providing technical advantages. The lower limit is that below which the  
20 rhodium and/or palladium have no observable effect for reducing the nitrogen oxide releases. This lower limit  
25 is generally about 100 ppm.

In the case of the dispersions obtained by the preparation method described below, it is conceivable  
30 for the rhodium and/or palladium to be also mainly present in the form of oxides or hydrated oxides. In the same case, the rhodium and/or palladium are moreover present in the dispersion essentially as being bound to the particles of the cerium compound. This  
35 bond between the element rhodium and/or palladium and the particles may be of a chemical type and it may be produced by the adsorption of said element on the particle surface.

The colloidal dispersion according to the invention comprises at least one acid, advantageously amphiphilic. The acid is more particularly selected from organic acids having at least 6 carbon atoms, even 5 more particularly 10 to 60 carbon atoms, preferably 15 to 25 carbon atoms.

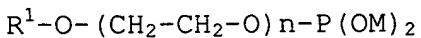
These acids may be linear or branched. They may be aryl, aliphatic or arylaliphatic acids, optionally containing other functional groups provided 10 that these functional groups are stable in the media in which the dispersions according to the present invention are to be used. Thus, it is possible to use, for example, aliphatic carboxylic acids, aliphatic sulfonic acids, aliphatic phosphonic acids, 15 alkylarylsulfonic acids and alkylarylphosphonic acids having about 10 to about 40 carbon atoms, whether natural or synthetic. It is obviously possible to use acid mixtures.

It is also possible to use carboxylic acids of 20 which the carbon chain bears ketone functional groups such as pyruvic acids substituted with the ketone functional group in the alpha position. They may also be alpha-halocarboxylic acids or alphahydroxycarboxylic acids. The chain attached to the carboxylic group may 25 carry unsaturated groups. In general, however, it is better to avoid too many double bonds because cerium catalyzes the crosslinking of double bonds. The chain may be interrupted by ether or ester functional groups provided that this does not excessively alter the 30 lipophilicity of the chain bearing the carboxylic group.

By way of example, mention can be made of fatty acids of tallol, soybean oil, soot, linseed oil, oleic acid, linoleic acid, stearic acid and isomers thereof, 35 pelargonic acid, capric acid, lauric acid, myristic acid, dodecylbenzenesulfonic acid, 2-ethylhexanoic acid, naphthenic acid, hexoic acid, toluenesulfonic acid, toluenephosphonic acid, laurylsulfonic acid,

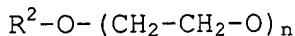
laurylphosphonic acid, palmitylsulfonic acid, and palmitylphosphonic acid.

In the context of the present invention, the term "amphiphilic acid" may also designate other 5 amphiphilic agents such as, for example, polyoxyethylene alkyl ether phosphates. The phosphates here are phosphates of the formula:



10

or polyoxyethylene dialkyl phosphates of the formula:



15

where:

20

- $R^1$ ,  $R^2$ ,  $R^3$ , identical or different, represent a linear or branched alkyl radical, particularly with 2 to 20 carbon atoms; a phenyl radical, an alkylaryl radical, more particularly an alkylphenyl radical, particularly with 8 to 12 carbon atoms; an arylalkyl radical, more particularly a phenylaryl radical;

25

- $n$  is the number of ethylene oxide possibly ranging from 0 to 12, for example;
- $M$  represents a hydrogen, sodium or potassium atom.

30

The radical  $R^1$  may be in particular a hexyl, octyl, decyl, dodecyl, oleyl or nonylphenyl radical.

35

By way of example of this type of amphiphilic compound, mention can be made of those marketed under the trademarks Lubrophoss® and Rhodafac® sold by Rhodia and particularly the following products:

- polyoxyethylene alkyl (C8-C10) ether phosphates Rhodafac® RA 600;
- polyoxyethylene tridecyl ether

phosphate Rhodafac® RS 710 or RS 410  
- polyoxyethylene oleocetyl ether  
phosphate Rhodafac® PA 35  
- polyoxyethylene nonylphenyl ether phosphate  
Rhodafac® PA 17  
- polyoxyethylene nonyl(branched) ether  
phosphate Rhodafac® RE 610.

The dispersions of the invention further comprise a liquid phase that is an organic phase and in which the particles are in suspension.

By way of example of an organic phase, mention can be made of aliphatic hydrocarbons such as hexane, heptane, octane, nonane, inert cycloaliphatic hydrocarbons such as cyclohexane, cyclopentane, cycloheptane, aromatic hydrocarbons such as benzene, toluene, ethylbenzene, xylenes, liquid naphthenes. Also suitable are petroleum cuts of the Isopar or Solvesso type (trademarks of Exxon), particularly Solvesso 100 which essentially contains a mixture of methyl ethylbenzene and trimethylbenzene, Solvesso 150 which contains a mixture of alkylbenzenes, particularly dimethylbenzene and tetramethylbenzene and Isopar which essentially contains C-11 and C-12 isoparaffinic and cycloparaffinic hydrocarbons. As other petroleum cuts, mention can also be made of those of the Petrolink® type marketed by Petrolink and of the Isane® type marketed by Total.

Chlorinated hydrocarbons can also be used for the organic phase, such as chlorobenzene or dichlorobenzene, and chlorotoluene. Ethers as well as aliphatic and cycloaliphatic ketones such as, for example, diisopropyl ether, dibutyl ether, methyl ethyl ketone, methyl isobutyl ketone, diisobutyl ketone, mesityl oxide, can be considered.

Esters may also be considered, but their drawback is the risk of hydrolysis. As ethers suitable for use, mention can be made of those produced by the reaction of acids with C1 to C8 alcohols, and

particularly palmitates of secondary alcohols such as isopropanol. By way of example, mention can also be made of butyl acetate.

Obviously, the organic phase may be based on a mixture of two or more hydrocarbons or compounds of the type described above.

The dispersions according to the invention have a concentration of cerium compounds, of the element rhodium and/or palladium and, optionally of the other rare earth and of iron, that generally may range up to 40% by weight of oxides of these elements with respect to the total weight of the dispersion. Above 40%, the viscosity of the dispersion is liable to be too high, particularly at low temperature. It is, however, preferable for this concentration to be at least 5%. Lower concentrations are economically less advantageous because of the volume of liquid phase which becomes too large.

The proportion between the organic phase and the acid or acids is not critical. The weight ratio between the organic phase and the acid or acids is selected preferably between 0.3 and 2.0.

The dispersion of the invention may be in a specific embodiment.

According to this embodiment, the dispersion is such that at least 90% of the particles are single-crystal particles. "Single-crystal" particles means particles which, when the dispersion is examined under a TEM (high-resolution transmission electron microscope), appear to be individualized and consisting of a single crystallite.

The cryo-TEM technique can also be used to determine the state of aggregation of the elementary particles. In this method, the transmission electron microscope (TEM) is used to observe samples that are kept frozen in their natural medium, which is either water or organic diluents such as aromatic or aliphatic solvents like, for example, Solvesso and Isopar, or

certain alcohols such as ethanol.

The frozen specimens are thin films about 50 to 100 nm thick frozen either in liquid ethane for aqueous samples or in liquid nitrogen for others.

5 With cryo-TEM, the state of dispersion of the particles is well preserved and representative of the state present in the real medium.

10 According to this embodiment, the particles have a fine and narrow particle size distribution. In fact, they have a  $d_{50}$  of between 1 and 5 nm, preferably between 2 and 4 nm.

15 In the present description, the particle size distribution properties refer to notations  $d_n$  where n is a number from 1 to 99. This notation represents the particle size such that n % by number of said particles have a size smaller than or equal to said size. For example, a  $d_{50}$  of 3 nanometers means that 50% by number of the particles have a size of 3 nanometers or smaller.

20 The particle size distribution is determined by conventional transmission electron microscopy (TEM), on a sample previously dried on a carbon membrane supported on a copper grid.

25 This preparation technique is preferred because it allows for better accuracy of particle size measurement. The zones selected for the measurements are those that have a state of dispersion similar to that observed by cryo-TEM.

30 The dispersion according to the specific embodiment given above can be prepared by a method comprising the following steps:

35 a) an aqueous mixture is prepared comprising at least one cerium salt, optionally a salt of a rare earth other than cerium and an iron salt, and a salt of at least one element selected from rhodium and palladium;

b) the aqueous mixture of step (a) is contacted with a basic medium to form a reaction mixture of which

the pH is maintained at a basic pH, thereby producing a precipitate;

5           c) the precipitate thus obtained is contacted with an acid and an organic phase, to obtain an organic colloidal dispersion.

10          The first step of the method (step a) consists in preparing an aqueous mixture, in the usual form of a solution or dispersion, of element(s) present in the composition of the particles to be obtained. This mixture comprises salts, preferably soluble, more particularly an acetate and/or a nitrate, of cerium and rhodium and/or palladium. In the case of the preparation of the dispersions according to the different variants described above, this mixture may 15 further comprise salts of other necessary elements, that is, salts of a rare earth other than cerium and/or an iron salt.

20          The next step (step b) consists in contacting the abovementioned aqueous mixture with a basic medium. Basic medium means any medium with a pH above 7. The 25 basic medium is normally an aqueous solution containing a base. Products of the hydroxide type can be used as a base in particular. Mention may be made of alkali metal or alkaline-earth metal hydroxides. Use may also be made of secondary, tertiary or quaternary amines. However, amines and ammonia may be preferred insofar as they decrease the risk of pollution by the alkali or alkaline-earth metal cations. Mention may also be made of urea.

30          The above mixture is contacted with the basic medium under conditions such that the pH of the reaction mixture formed remains basic. Thus, the pH of the reaction mixture is maintained at a value of at least 7, more particularly of at least 7.5 and even 35 more particularly of between 7.5 and 11.

The aqueous mixture can be contacted with the basic medium by introducing the above mixture into the basic medium. Contact can be continuous, provided that

the pH is controlled by adjusting the respective flow rates of the mixture and the basic medium.

According to a particular embodiment of the invention, it is possible to operate in conditions such 5 that when the mixture is contacted with the basic medium, the pH of the reaction medium thus formed is kept constant. Constant pH means a pH variation of not more than  $\pm 0.2$  pH units from the set value. Such 10 conditions can be obtained by introducing an additional quantity of base into the reaction mixture formed, during the introduction of the mixture into the basic medium.

The contacting is normally carried out at ambient temperature. This contacting can 15 advantageously be obtained under an atmosphere of air or nitrogen or a nitrogen-air mixture.

A precipitate is recovered on completion of the reaction.

This precipitate can optionally be separated 20 from its mother liquor by filtration, centrifugation or any other means known to a person skilled in the art for such a separation. The separated product can be washed.

Preferably, the precipitate is left in wet 25 form, that is, it is not subjected to a drying or a freeze-drying step or any operation of this type.

In the rest of the method, the precipitate can be used as such, or optionally after being placed again in aqueous suspension.

30 The precipitate is then contacted with at least one acid and one organic phase, as defined above (step c).

During step (c), the precipitate is used in its 35 wet form, and the proportion of oxides of cerium, of the other element rhodium and/or palladium and optionally of another rare earth and/or iron, of said precipitate may vary between 10 and 50% by weight of the weight of the wet precipitate. The percentages of

total oxides can be determined by loss on ignition, for example, by calcination at 1000°C.

To obtain an organic colloidal dispersion in step (c), the optionally redispersed precipitate is 5 contacted with at least one acid and one organic phase like those described above. The quantity of acid to be incorporated may be defined by the molar ratio r: number of moles of acid/number of moles of cerium and/or optionally of the other rare earth and/or of 10 iron.

This molar ratio may be between 0.2 and 0.8, preferably between 0.3 and 0.6.

The quantity of organic phase to be incorporated is adjusted in order to obtain a total 15 oxide concentration such as that mentioned above.

At this stage, it may be advantageous to add a promoter to the organic phase, its function being to accelerate the transfer of the particles of compound(s) from the aqueous phase to the organic phase and to 20 improve the stability of the organic colloidal dispersions obtained.

Promoters that are suitable include compounds with an alcohol function and particularly linear or branched aliphatic alcohols with 6 to 12 carbon atoms. 25 By way of specific examples, mention can be made of 2-ethylhexanol, decanol, dodecanol or mixtures thereof.

The proportion of said agent is not critical and may vary within wide limits. However, a proportion of between 2 and 15% by weight is generally suitable.

The order of introduction of the various 30 reagents is immaterial. The precipitate or its aqueous suspension, the acid, the organic phase, and optionally the promoter, can be mixed simultaneously. The acid, the organic phase and optionally the promoter can also 35 be premixed.

The aqueous colloidal dispersion can be contacted with the organic phase in a reactor that is under an atmosphere of air, nitrogen or an air-nitrogen

mixture.

Although the aqueous colloidal dispersion can be contacted with the organic phase at ambient temperature, about 20°C, it is preferable to operate at 5 a temperature selected in an interval ranging from 60°C to 150°C, advantageously between 80°C and 140°C.

In certain cases, due to the volatility of the organic phase, it is necessary to condense its vapors by cooling to a temperature below its boiling point.

10 The resulting reaction mixture (mixture of aqueous colloidal dispersion, acid, organic phase and optionally promoter) is maintained with stirring throughout the duration of the heating, which may vary.

When the heating is stopped, the presence of 15 two phases is observed: an organic phase containing the colloidal dispersion, and a residual aqueous phase.

The presence of a third emulsified phase may sometimes be observed.

20 The organic phase and the aqueous phase are then separated by conventional separation techniques: settling, centrifugation.

As indicated above, the method described 25 applies to the preparation of a dispersion according to the abovementioned specific embodiment. It is also possible to implement a method different from the one given above by the fact that it comprises, between steps (b) and (c), a drying of the precipitate particularly by spray drying or freeze drying. This method leads to a dispersion according to the invention but the particles of the specific embodiment do not 30 have the characteristic of being single crystals.

The organic colloidal dispersions described above can be employed as additives for motor fuels, particularly diesel, for internal combustion engines, 35 more particularly as a diesel fuel additive for a diesel engine.

Finally, the invention relates to a fuel for internal combustion engines that contains a colloidal

dispersion of the type described above. This fuel is obtained by mixing a standard fuel, particularly of the diesel type, with the dispersion of the invention, generally in a proportion such that the ratio of element Ce + element Rh and/or Pd metal + optionally the rare earth element and iron, to the mass of fuel, is between 5 and 200 ppm.

The presence of the dispersions of the invention in fuels has the effect of lowering the autoignition temperature of the soot and reducing the emission of nitrogen oxides in the engine exhaust gases, and it can also contribute to the oxidation of the carbon monoxide and unburnt hydrocarbons.

Examples will now be provided.

15

#### EXAMPLE 1

This example relates to the preparation of a dispersion according to the invention, based on cerium, iron and rhodium. This preparation is made in two steps: the first leads to the formation of a solid precipitate in the aqueous phase, and the second relates to the transfer of this precipitate to the organic phase.

1) Synthesis of a solid precipitate in aqueous  
25 phase

a) Preparation of an iron acetate solution  
206.1 g of ferric (III) nitrate nonahydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) of 98% purity from Prolabo) is dissolved in 1 l of purified water in order to prepare a solution containing 0.5 mol/l of Fe. 270 ml of a 10% by volume ammonia solution is added to the ferric nitrate solution thus prepared and stirred, using a peristaltic pump, at the rate of 10 ml/min.

The neutral pH suspension obtained is  
35 centrifuged at 4500 rpm for 12 minutes. The precipitate recovered is replaced in suspension of the initial volume with purified water. The suspension is stirred for 15 minutes, the precipitate is separated

again under the same conditions, and again replaced in suspension with an equivalent final volume.

A dispersion with pH 6.5 is thereby obtained, to which 100 ml of acetic acid ( $\text{CH}_3\text{COOH}$  100% from Prolabo) is added, to yield an iron acetate solution with pH 2.7 and oxide concentration of 2.8% of  $\text{Fe}_2\text{O}_3$  (determined by loss on ignition).

b) Preparation of a coacetate solution

139.7 g of crystallized cerium (III) acetate ( $\text{Ce}(\text{CH}_3\text{COO})_3$  from Rhodia Electronics and Catalysis comprising 49.29% of oxide  $\text{CeO}_2$ ) is dissolved in 0.8 l of purified water. 6.7 g of acetic acid ( $\text{CH}_3\text{COOH}$  100% from Prolabo), 3 g of a rhodium (III) nitrate solution ( $\text{Rh}(\text{NO}_3)_3$  concentrated to 10% of Rh from Aldrich) and 1124.6 g of the previously prepared iron acetate solution, are added to this solution. The mixture is made up to 2.5 l with purified water.

c) Precipitation of the coacetate solution

The precipitation is carried out in a continuous assembly comprising:

- a 1 l reactor equipped with a propeller stirrer, set at 400 rpm, with a stock of 0.5 l of basic solution ( $\text{NH}_4\text{OH}$  at pH=10.5) and an electrode servocontrolled by a pH control pump with a setpoint at pH 10.5;

- two feed bottles, one containing the coacetate solution previously prepared and the other a 6N ammonia solution. The flow rate of the coacetate solution is set at 500 ml/h and the flow rate of ammonia is servocontrolled by the pH regulation;

- a drawoff system that is used to adjust the volume in the reactor to 0.5 l and connected to a second reactor placed in series with the first;

- a second reactor for recovering the precipitate formed.

The precipitate is recovered by centrifugation at 3000 rpm for 12 minutes and then replaced in suspension in purified water in a concentration of 50

g/l of total oxide.

2) Transfer to organic phase

5        340 ml of the previously described aqueous suspension is placed in a 2 liter double jacket reactor equipped with a controlled temperature bath. An organic phase containing 129.9 g of Isopar L (paraffinic solvent from Exxon) and 23.1 g of isostearic acid (Prisorine 3501 solution from Uniquema) 10 previously dissolved, are added at ambient temperature.

The two-phase mixture is then heated to 95°C in 1h 30 min, with the stirrer speed set at 220 rpm. The mixture is kept at 95°C for 4 hours, and then left to cool at ambient temperature. The coalescence obtained 15 when stirring is stopped reveals the formation of a black organic phase, above a clear aqueous phase. The oxide concentration of the aqueous phase, determined by loss on ignition, is negligible, attesting to a quantitative transfer.

20

3) Characterization of the organic colloidal dispersion based on cerium, iron and rhodium.

The concentration of the organic colloidal phase, determined after evaporation of the Isopar L and 25 calcination at 1000°C, is equal to 9.9% of total oxide. The composition of the oxide dispersed in the organic phase, determined by potentiometric methods (Ce) and polarographic methods (Fe), is equimolar in Ce/Fe and contains 0.26% of rhodium (mass of rhodium metal/mass 30 of elemental cerium and iron), the rhodium being determined by ICP/OES. Analysis by cryo-transmission electron microscopy of the organic colloidal phase reveals perfectly individualized particles with a diameter of 3 to 5 nm.

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EXAMPLE 2

This example relates to the evaluation on an engine test bench of the product obtained in the

previous example.

5       The evaluation is made using a Peugeot 2.2 l cubic capacity diesel engine, reference DW 12 TED4/L4, placed on a dynamometric test bench. The exhaust line is equipped with a silicon carbide particulate filter produced by Ibiden (5.66 x 6200 cpsi).

10      The dispersion of the invention is added as an additive to a diesel containing 7 ppm of sulfur to obtain a proportion of 10 ppm (Ce+Fe+Rh metal) with respect to the diesel containing additive.

15      The particulate filter is then loaded under the following conditions:

- engine speed: 3000 rpm
- torque: 30 Nm
- loading time: 10 hours.

During this loading phase, the nitrogen oxide NO and NO<sub>2</sub> emissions are measured continuously using an Ecophysic CLD 700 analyzer.

20      The results obtained are given below. The comparative test is the test performed with, as the additive, a dispersion prepared following the procedure of example 1 but without rhodium.

Test	NO + NO <sub>2</sub> emissions measured at end of filter loading (t = 10 h) in ppm
Example 2	100
Comparative	125

25      A 20% reduction of nitrogen oxide releases is observed when the dispersion according to the invention is used.

All the soot accumulated in the filter during 10 hours is then burned at 500°C in 320 seconds.